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• **Comments:**

In re Application of: Guido VOIT et al.
U.S. Patent No.: 6,852,669
Serial No.: 09/851,214
Issue Date: February 8, 2005
Title: HYDROGENATION CATALYST
Attachments: Request for Certificate of Correction
Certificate of Correction Form PTO/SB/44 (PTO-1050)
Mark-up copy of Letters Patent

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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Guido VOIT et al.

Art Unit: 1626

Patent No.: 6,852,669

Examiner: Ebenezer O. Sackey

Issued: February 8, 2005

Confirmation No.: 4235

For: HYDROGENATION CATALYST

Attorney Docket: 48839DIV

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Commissioner for Patents
P.O. Box 1450
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REQUEST FOR CERTIFICATE OF CORRECTION

Sir:

Applicants herewith submit a Certificate of Correction Form PTO/SB/44. It is respectfully requested that the Certificate of Corrections be entered.

The changes noted on the Certificate of Correction Form PTO/SB/44 correct the errors which occurred on the part of the U.S. Patent and Trademark Office. No fee should therefore be required.

It is not believed that a fee is required for filing of this paper. However, please charge any shortage in fees due in connection with the filing of this paper to Deposit Account No. 14.1437. Please credit any excess fees to such deposit account.

Respectfully submitted,

By: Jason D. Volght

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Date: April 28, 2006

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Michelle Bryant
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PTO/SB/44 (04-05)

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**UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION**Page 1 of 1

PATENT NO. : 6,852,669
APPLICATION NO. : 09/851,214
ISSUE DATE : February 8, 2005
INVENTOR(S) : Guido VOIT et al.

It is certified that an error appears or errors appear in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Claim 1, column 7, line 52, "to It by" should read --to 1% by--.

Claim 6, column 7, line 67, "m²/g" should read --m²/g--.

Claim 18, column 8, line 58, "DET" should read --BET--.

MAILING ADDRESS OF SENDER (Please do not use customer number below):

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PAGE 3/4 * RCVD AT 5/11/2006 11:07:13 AM [Eastern Daylight Time] * SVR:USPTO-EFXRF-1/2 * DNIS:2738300 * CSID:2026590105 * DURATION (mm-ss):02-10

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US 6,852,669 B2

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used had the following composition: 72% by weight of Fe, 0.06% by weight of Al, 0.03% by weight of Ca, 0.04% by weight of Mg, 0.10% by weight of Si, 0.01% by weight of Ti, 0.13% by weight of Mn, remainder oxygen.

The cooled melt block was comminuted in a jaw crusher, and a sieve fraction of particle size 1.5-3 mm was separated out by sieving. The oxidic catalyst was reduced in an H_2/N_2 stream at 450° C. for 72 hours. After cooling down to room temperature under nitrogen, the Fe catalyst was passivated with an N_2 /air stream (24 hours with 1% of air in nitrogen), care being taken to ensure that the temperature in the catalyst bed did not rise above 45° C.

b) Hydrogenation of ADN to HMD and/or ACN

Three serially connected tubular reactors (total length 4.5 m, d=6 mm) were packed with 142 mL (240 g) of the catalyst (particle size range from 1.5 to 3 mm) prepared according to Example 1 a) and then reduced in a 200 L/h stream of hydrogen at atmospheric pressure. To this end, the temperature was raised from 70° C. to 340° C. over 24 hours and subsequently held at 340° C. for 72 hours. After the temperature had been lowered, the reactor was fed with a mixture of 74 or 148 mL/h of ADN (catalyst space velocity 0.5 or 1.0 kg of ADN/L of cat./h), 365 mL/h of NH_3 and 200 standard L/h of H_2 at 250 bar. No decrease in catalyst activity was observed after a run of 7000 hours. Under the conditions recited in Table 1, the following results were obtained as a function of the temperature and the catalyst space velocity (Table 1):

Hexamethylenediamine by hydrogenation of adiponitrile

Temperature (° C.)	Pressure (bar)	Cat. space velocity (kg)	ADN conversion	HMD selectivity	ACN	ICCP	AMCPA	DCH	THA
115	250	0.5	100	99.0	100	55	24	1150	30
135	250	1.0	100	98.9	110	60	35	1800	43
98	250	0.5	80	42.3	56.8 ¹⁾	94	41	1130	

¹⁾ ACN selectivity (%)

We claim:

1. A hydrogenation catalyst comprising, as catalytically effective component, a composition consisting of

(a) iron or a compound based on iron or a mixture thereof,

(b) from 0.001 to 0.3% by weight based on (a) of a promoter based on 2, 3, 4 or 5 elements selected from the group consisting of aluminum, silicon, zirconium, titanium and vanadium,

(c) from 0 to 0.3% by weight based on (a) of a compound based on an alkali and/or alkaline earth metal, and

(d) from 0.001 to 1% by weight based on (a) of manganese.

2. The catalyst defined in claim 1, wherein the catalytically effective component is obtained by reduction with or without subsequent passivation of a magnetite.

3. The catalyst defined in claim 1, wherein the catalytically effective component is obtained by precipitating precursors of constituents (a), (b), (d) and optionally (c) in the presence or absence of support materials.

4. The catalyst defined in claim 1, which is obtained by impregnating a support with a solution of constituents (a), (b), (d) and optionally (c).

5. The catalyst defined in claim 1, which is obtained by spraying constituents (a), (b), (d) and optionally (c) onto a support.

6. The catalyst defined in claim 1, which has a BET surface area of from 3 to 20 m²/g, a total pore volume of

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from 0.05 to 0.2 mL/g, an average pore diameter of from 0.03 to 0.1 μ m and a 0.01 to 0.1 μ m pore volume fraction within the range from 50 to 70%.

7. The catalyst defined in claim 1, wherein the promoter elements (b) are selected from aluminum, silicon and titanium.

8. The catalyst defined in claim 1, wherein constituent (c) is based on magnesium and/or calcium.

9. The catalyst defined in claim 1, wherein constituent (c) is present in an amount of from 0.01 to 0.2% by weight based on (a).

10. The catalyst defined in claim 1, wherein constituent (c) is present in an amount of from 0.01 to 0.1% by weight based on (a).

11. The catalyst defined in claim 1, wherein constituent (d) is present in an amount of from 0.001 to 0.3% by weight based on (a).

12. The catalyst defined in claim 1, wherein constituent (d) is present in an amount of from 0.01 to 0.2% by weight based on (a).

13. A hydrogenation catalyst consisting essentially of a catalytically effective component and a support material wherein the catalytically effective component is a composition consisting of

(a) iron or a compound based on iron or a mixture thereof,

(b) from 0.001 to 0.3% by weight based on (a) of a promoter based on 2, 3, 4 or 5 elements selected from the group consisting of aluminum, silicon, zirconium, titanium and vanadium,

(c) from 0 to 0.3% by weight based on (a) of a compound based on an alkali and/or alkaline earth metal, and

(d) from 0.001 to 1% by weight based on (a) of manganese.

14. The catalyst defined in claim 13, wherein the catalytically effective component is obtained by reduction with or without subsequent passivation of a magnetite.

15. The catalyst defined in claim 13, which is obtained by precipitating precursors of constituents (a), (b), (d) and optionally (c) in the presence of the support materials.

16. The catalyst defined in claim 13, which is obtained by impregnating the support with a solution of constituents (a), (b), (d) and optionally (c).

17. The catalyst defined in claim 13, which is obtained by spraying constituents (a), (b), (d) and optionally (c) onto the support.

18. The catalyst defined in claim 13, which has a BET surface area of from 3 to 20 m²/g, a total pore volume of from 0.05 to 0.2 mL/g, an average pore diameter of from 0.03 to 0.1 μ m and a 0.01 to 0.1 μ m pore volume fraction within the range from 50 to 70%.

19. The catalyst defined in claim 13, wherein constituent (c) is present in an amount of from 0.01 to 0.2% by weight based on (a).

20. The catalyst defined in claim 13, wherein constituent (d) is present in an amount of from 0.001 to 0.3% by weight based on (a).

21. The catalyst defined in claim 13, wherein constituent (d) is present in an amount of from 0.01 to 0.2% by weight based on (a).

22. The catalyst defined in claim 13, which has a BET surface area of from 3 to 20 m²/g, a total pore volume of from 0.05 to 0.2 mL/g, an average pore diameter of from 0.03 to 0.1 μ m and a 0.01 to 0.1 μ m pore volume fraction within the range from 50 to 70%.

23. The catalyst defined in claim 13, wherein constituent (c) is present in an amount of from 0.01 to 0.2% by weight based on (a).

24. The catalyst defined in claim 13, wherein constituent (d) is present in an amount of from 0.001 to 0.3% by weight based on (a).

25. The catalyst defined in claim 13, wherein constituent (d) is present in an amount of from 0.01 to 0.2% by weight based on (a).

26. The catalyst defined in claim 13, which has a BET surface area of from 3 to 20 m²/g, a total pore volume of from 0.05 to 0.2 mL/g, an average pore diameter of from 0.03 to 0.1 μ m and a 0.01 to 0.1 μ m pore volume fraction within the range from 50 to 70%.

27. The catalyst defined in claim 13, wherein constituent (c) is present in an amount of from 0.01 to 0.2% by weight based on (a).

28. The catalyst defined in claim 13, wherein constituent (d) is present in an amount of from 0.001 to 0.3% by weight based on (a).